

Crystal and Molecular Structure and Spectra of 2',2',5,5,5',5',7,7-Octamethyl-2',5,5',7-tetrahydrofuro[3,4-*d*]-1-oxa-3,4-diselenin-2-spiro-3'-furan-4'(3'*H*)-one from Selenium Dioxide Oxidation of 2,2,5,5-Tetramethyltetrahydrofuran-3-one. A Novel Six-membered Heterocyclic Ring System in the Chalcogen Series

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Received November 30, 1992

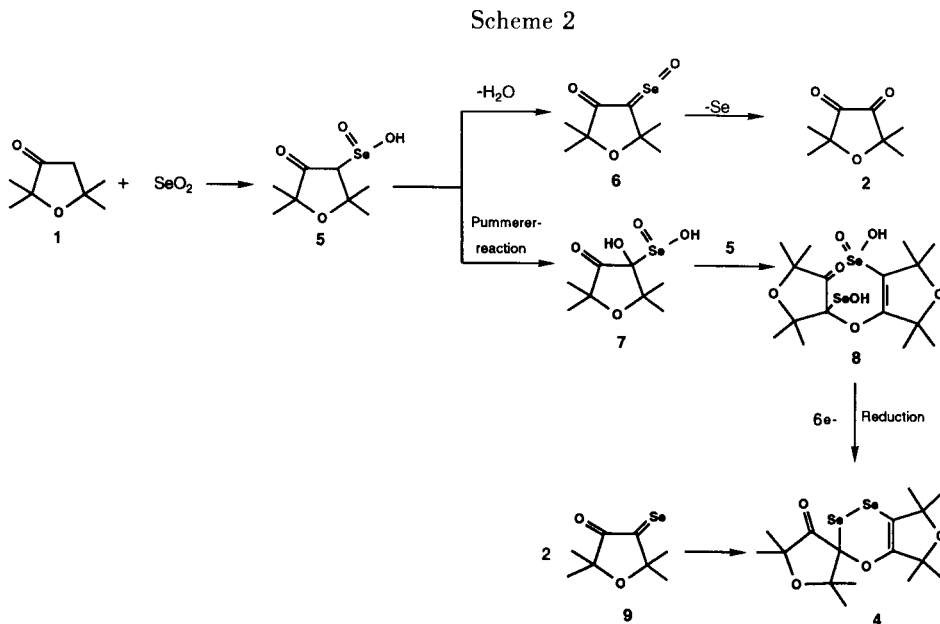
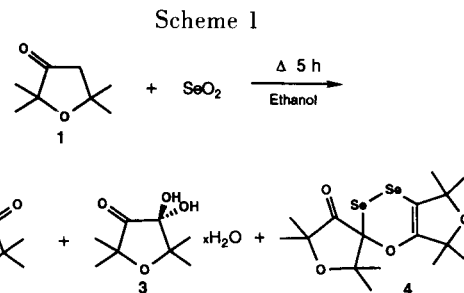
When 2,2,5,5-tetramethyltetrahydrofuran-3-one **1** was oxidized with selenium dioxide in ethanol, the title heterocycle **4** was formed along with 2,2,5,5-tetramethyltetrahydro-3,4-furandione **2**. The diselenide **4** represents a novel six-membered heterocycle in the chalcogen series.

J. Heterocyclic Chem., **30**, 961 (1993).

Formation of organic selenium compounds in selenium dioxide oxidations of activated C-H bonds and aromatics occasionally occurs, especially with multifunctional substrates [1-5]. We have recently reported the syntheses and structures of organic selenium compounds formed in selenium dioxide oxidations [6-8]. In order to prepare 2,2,5,5-tetramethyltetrahydro-3,4-furandione **2** from 2,2,5,5-tetramethyltetrahydrofuran-3-one **1** by selenium dioxide oxidation, the dione **2**, its dihydrate **3** and heterocycle **4** were formed in ethanol (Scheme 1).

Solvents modify the actual oxidizing agent, and also here ethanol and 1,4-dioxane gave a slightly different product ratio. Since the oxidation of 2,2,4,4-tetramethylcyclopentanone does not give the heterocycle analogous to **4**,

the presence of the electronegative oxygen atom in **1** affects the reaction path postulated in Scheme 2. The only reactive position in **1** gives the intermediate **5** on the basis of the mechanism [9]. This reacts *via* dehydration to



selenine **6**, which rearranges to **2** with repulsion of the reduced selenium. Because in the formation of **4**, there is a total of 6 electron reduction in selenium and only 2 electron oxidation in **1**, one must assume redox reactions not to be seen among isolated products. For the formation of **8**, a reaction between Pummerer-product **7** and **5** or the anhydride of **5**, the selenine **6**, *via* elimination of water seems reasonable based on earlier data [7]. The final step, the formation of the diselenide bridge, necessitates two two-electron reductions at selenium. Due to the disproportionation reactions and equilibria involved in Se-acids, there are many available pathways to the formation of **4** [10]. One simple pathway is a dimerization of a selenoketone **9** formed in a redox reaction of selenine **6** analogously to that observed with α -oxothioketone [11-13].

Molecular Structure and Spectral Data of **4**.

A high resolution ms of **4** established its molecular formula as $C_{16}H_{24}O_4Se_2$. The features related to the function-

Table I

Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Coefficients ($\text{\AA}^2 \times 10^3$) for Non-hydrogen Atoms of **4** with Standard Deviation in Parentheses [a]

Atom	x	y	z	U(eqv) [b]
Se(1)	198(1)	-1211(1)	1795(1)	50(1)
Se(2)	-2567(1)	72(1)	661(1)	51(1)
C(1)	-1893(11)	-1871(5)	2776(4)	37(2)
O(1)	-3312(8)	-868(3)	3066(3)	45(2)
C(2)	-4282(11)	336(5)	2467(4)	41(2)
C(3)	-4164(12)	871(6)	1534(4)	46(3)
C(4)	-5460(3)	2286(6)	1285(4)	53(3)
C(41)	-7416(15)	2583(8)	604(5)	91(4)
C(42)	-3997(17)	3217(7)	933(6)	91(4)
O(2)	-6273(9)	2460(4)	2178(3)	61(2)
C(5)	-5511(12)	1293(5)	2966(4)	44(3)
C(51)	-4083(13)	1571(7)	3643(5)	60(3)
C(52)	-7509(12)	896(7)	3446(5)	58(3)
C(6)	-544(11)	-2895(6)	3669(4)	39(2)
C(61)	1506(13)	-2606(8)	3957(5)	71(4)
C(62)	-2076(13)	-3067(6)	4514(4)	59(3)
O(4)	79(8)	-4067(4)	3359(3)	52(2)
C(8)	-2903(12)	-2697(6)	2349(4)	41(3)
O(3)	-4581(8)	-2345(5)	1872(3)	59(2)
C(7)	-1499(14)	-4123(6)	2689(5)	57(3)
C(71)	-247(16)	-4558(7)	1874(5)	84(4)
C(72)	-2872(16)	-5053(7)	3143(6)	88(4)

[a] For atomic labelling, see Figure 2. [b] Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

Table II

Bond lengths (\AA) in **4** with Standard Deviations in Parentheses

Se(1)-Se(2)	2.330(2)	C(4)-O(2)	1.436(8)
Se(1)-C(1)	2.025(6)	O(2)-C(5)	1.451(6)
Se(2)-C(3)	1.885(7)	C(5)-C(51)	1.500(11)
C(1)-O(1)	1.402(7)	C(5)-C(52)	1.523(11)
C(1)-C(6)	1.570(7)	C(6)-C(61)	1.504(12)
C(1)-C(8)	1.502(11)	C(6)-C(62)	1.546(9)
O(1)-C(2)	1.371(6)	C(6)-O(4)	1.452(8)
C(2)-C(3)	1.334(8)	O(4)-C(7)	1.441(10)
C(2)-C(5)	1.500(9)	C(8)-O(3)	1.202(9)
C(3)-C(4)	1.519(8)	C(8)-C(7)	1.551(8)
C(4)-C(41)	1.520(12)	C(7)-C(71)	1.536(12)
C(4)-C(42)	1.521(13)	C(7)-C(72)	1.504(13)

alities of **4** were clarified by means of its spectral data: uv, ir, nmr (1H , ^{13}C , ^{77}Se), and ms. The molecular structure could be unequivocally assigned by a single crystal X-ray analysis.

Crystal and Molecular Structure of **4**.

The final atomic coordinates and equivalent isotropic displacement coefficients for non-hydrogen atoms of **4** are given in Table I. The bond lengths, bond angles and deviations of atoms from some selected least-squares planes are presented in Tables II, III and IV, respectively.

Table III

Bond Angles ($^\circ$) in **4** with Standard Deviations in Parentheses

Se(2)-Se(1)-C(1)	94.3(2)	Se(1)-Se(2)-C(3)	93.1(2)
Se(1)-C(1)-O(1)	112.7(4)	Se(1)-C(1)-C(6)	109.2(4)
O(1)-C(1)-C(6)	109.2(5)	Se(1)-C(1)-C(8)	104.7(4)
O(1)-C(1)-C(8)	117.6(6)	C(6)-C(1)-C(8)	102.8(5)
C(1)-O(1)-C(2)	124.4(4)	O(1)-C(2)-C(3)	132.5(6)
O(1)-C(2)-C(5)	114.2(5)	C(3)-C(2)-C(5)	112.9(5)
Se(2)-C(3)-C(2)	127.1(4)	Se(2)-C(3)-C(4)	124.5(4)
C(2)-C(3)-C(4)	108.3(5)	C(3)-C(4)-C(41)	113.1(7)
C(3)-C(4)-C(42)	111.7(6)	C(41)-C(4)-C(42)	112.1(6)
C(3)-C(4)-O(2)	104.3(4)	C(41)-C(4)-O(2)	108.0(6)
C(42)-C(4)-O(2)	107.1(6)	C(4)-O(2)-C(5)	112.2(4)
C(2)-C(5)-O(2)	102.1(4)	C(2)-C(5)-C(51)	113.1(6)
C(2)-C(5)-C(51)	109.2(5)	C(2)-C(5)-C(52)	112.1(6)
C(2)-C(5)-C(52)	108.1(5)	C(51)-C(5)-C(52)	111.7(6)
C(1)-C(6)-C(61)	116.2(5)	C(1)-C(6)-C(62)	108.4(5)
C(61)-C(6)-C(62)	110.1(6)	C(1)-C(6)-O(4)	102.4(5)
C(61)-C(6)-O(4)	108.2(5)	C(62)-C(6)-O(4)	111.1(5)
C(6)-O(4)-C(7)	112.9(5)	C(1)-C(8)-O(3)	126.6(6)
C(1)-C(8)-C(7)	108.3(6)	O(3)-C(8)-C(7)	125.0(7)
O(4)-C(7)-C(8)	103.3(6)	O(4)-C(7)-C(71)	108.0(7)
C(8)-C(7)-C(71)	112.1(5)	O(4)-C(7)-C(72)	112.1(6)
C(8)-C(7)-C(72)	111.5(7)	C(71)-C(7)-C(72)	109.7(7)

Table IV

Deviations (\AA) of Atoms from Selected Least-Squares Planes

Plane 1. Plane through C(2), C(3) and C(4)						
Atom	Se(1)	Se(2)	C(1)	O(1)	O(2)	C(5)
Dev.	1.4136	0.1006	0.2000	0.0722	-0.0065	0.0473
Atom	C(6)	O(4)	C(8)	O(3)	C(7)	
Dev.	0.7242	0.2704	-1.0762	-2.0358	-0.9633	
Plane 2. Plane through C(1), O(1) and C(2)						
Atom	Se(1)	Se(2)	C(3)	C(4)	O(2)	C(5)
Dev.	1.1518	-0.0753	-0.0226	0.0970	0.1804	0.1811
Atom	C(6)	O(4)	C(8)	O(3)	C(7)	
Dev.	0.4764	-0.1006	-1.3414	-2.2666	-1.3454	
Plane 3. Plane through C(1), C(6) and O(4)						
Atom	Se(1)	Se(2)	O(1)	C(2)	C(3)	C(4)
Dev.	1.8559	1.2126	-0.5347	-0.3478	0.2862	0.2327
Atom	O(2)	C(5)	C(8)	O(3)	C(7)	
Dev.	-0.5097	-0.8631	-0.7922	-1.4341	-0.6890	
Plane 4. Plane through Se(1), Se(2), and C(1)						
Atom	O(1)	C(2)	C(3)	C(4)	O(2)	C(5)
Dev.	1.1454	1.7304	1.4451	2.5380	3.4451	3.0432
Atom	C(6)	O(4)	C(8)	O(3)	C(7)	
Dev.	-0.0895	-1.5074	-1.3299	-1.5661	-2.3303	

The unit-cell is shown in Figure 1, and the layer-like nature is emphasized. The crystal structure consists of non-interacting molecules except the interaction between

Se(1) and O(3) (3.216 Å), which confers orientational demands on the crystal packing of the molecules.

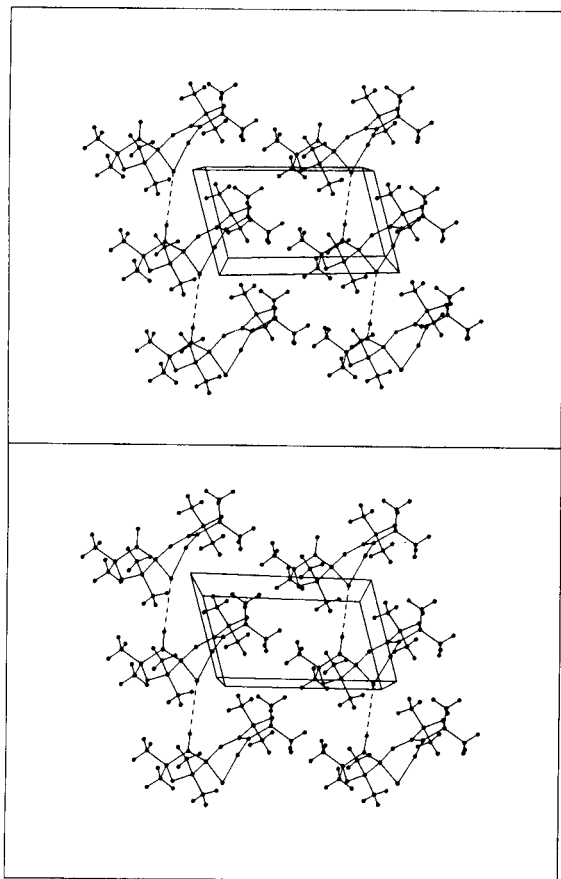


Figure 1. Stereoscopic View of the Molecular Packing in the Unit-Cell of **4**.

An illustration of the molecular structure of **4** with the numbering scheme is given in Figure 2. The structural parameters in the heterocycle are those found in 1,3-oxaselenole derivatives [7] and fall within the average values [14]. Expectedly bond angles at selenium in **4** are larger

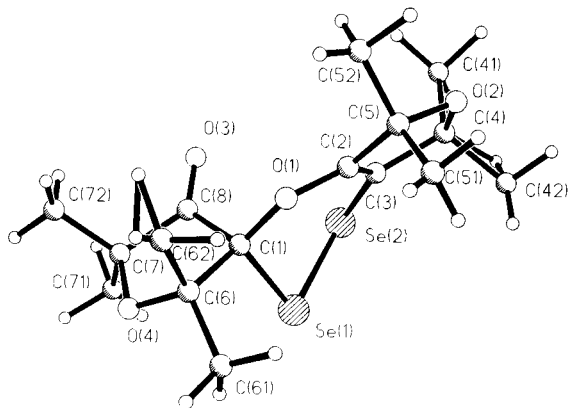


Figure 2. Molecular Structure and Crystallographic Numbering Scheme Employed for $C_{16}H_{24}O_4Se_2$.

than in the five-membered rings in 1,3-oxaselenoles [7]. The structural parameters in the furane rings are the same within the experimental error as those found in a related compound [15]. The conformation of the Se-Se-heterocycle is an half-chair Se(1) deviating 1.1518 Å from the plane defined by C(1)-O(1)-C(2) (Table IV). The reason for the large deviation of Se(1) from the plane C(1)-O(1)-C(2) in the solid state may be caused by intermolecular attraction between Se(1) and O(3). Both furane rings are nearly planar, the largest deviation from the plane defined by C(2)-C(3)-C(4) is that of C(5) (0.0473 Å) and from the plane defined by C(1)-C(6)-O(4) that of C(8) (-0.7922 Å). The fusion *via*

Table V
Experimental Crystal Data and
Structure Refinement Procedures for **4**

Crystal data	
Formula	$C_{16}H_{24}O_4Se_2$
Formula weight	438.3
Crystal size (mm)	0.3 x 0.4 x 0.4
Crystal system	Triclinic
Space group	P-1
Unit cell dimensions	$a = 6.308(5) \text{ \AA}$ $b = 11.034(4) \text{ \AA}$ $c = 14.642(3) \text{ \AA}$ $\alpha = 73.55(3)^\circ$ $\beta = 88.33(4)^\circ$ $\gamma = 75.76(4)^\circ$
Volume	946.5(8) \AA^3
Z	2
Density (Calcd.)	438.3 Mg/m^3
Absorption coefficient	3.920 mm^{-1}
F(000)	440
Experimental data	
Diffractometer used	Nicolet P3F
Radiation	MoK α ($\lambda = 0.71069 \text{ \AA}$)
Temperature	23 $^\circ$
Monochromator	Highly oriented graphite crystal
2 θ range	2.0 to 48.0 $^\circ$
Scan type	ω
Scan speed	Variable; 1.50 to 29.30 $^\circ$ / minute in ω
Scan range (ω)	1.00 $^\circ$
Background measurement	Stationary crystal and stationary counter at beginning and end of scan, each for 25.0% of total scan time
Standard reflections	2 measured every 98 reflections
Index ranges	0 $\leq h \leq 8$, -13 $\leq k \leq 14$, -19 $\leq l \leq 19$
Reflections collected	4743
Independent reflections	4743 ($R_{\text{int}} = 0.00\%$)
Observed reflections	2954 [$F > 6.0\sigma(F)$]
Solution and refinement	
System used	Siemens SHELXTL PLUS (PC Version)
Solution	Direct methods
Refinement method	Full-Matrix Least-Squares
Quantity minimized	$\Sigma w(F_o - F_c)^2$
Hydrogen atoms	Riding model, fixed isotropic U
Weighting scheme	$w^{-1} = \delta^2(F) + 0.000F^2$
Number of parameters refined	199
Final R indices (obs Data)	R = 4.84%, wR = 5.30%

the C=C double bond makes the two rings nearly planar, and the other furane ring plane is nearly perpendicularly positioned. At the spiro carbon atom C(1) the angle between the planes defined by Se(1)-Se(2)-C(1) and C(1)-C(6)-O(4) is 80.4 $^\circ$.

EXPERIMENTAL

Melting points were determined with an Electrothermal apparatus and are uncorrected. The analytical purity checks were done with a Carlo Erba Fractovap G1 gas chromatograph using SE-52 or FFAP capillary columns (30 m/0.30 mm i.d.), flame ionization detector, and temperature program 50-230°/10°/minute and with a DuPont Model 850 HPLC system with Rheodyne 7120 injector, DuPont variable wavelength uv detector (190-350 nm), and a Spectra-Physics Minigrator integrator together with a Goerz Servogro recorder. The hplc analyses were performed using a methanol-water or acetonitrile-water solvent system under isocratic and gradient conditions with LiChrosorb Hibar RP-18 column, 5 μ m, 250 x 4.0 mm (from Merck). The column temperature was 35 and 50°. The flow rate was 1.0 ml/minute. The detector wavelength was 240-300 nm. Ultraviolet spectra were obtained with a Shimadzu UV-200 double beam spectrophotometer using quartz cells with a 1.00 cm light path. Infrared spectra were recorded on a Perkin-Elmer 125 or Nicolet 20 x cs FT-IR instruments. Mass spectra were obtained on a JEOL JMS-SX 102 instrument, with a direct sample probe and an ionization energy of 70 eV at a source temperature of about 50°. Elemental composition and exact mass measurement were performed at a resolving power of 10000. The ¹H nmr and ¹³C nmr spectra were run on a JEOL JNM-PMX60, JEOL JNM-PFT100, and Varian Gemini-200 instruments. Chemical shifts are reported in ppm relative to tetramethylsilane. The ⁷⁷Se nmr spectra were recorded on a JEOL FX-100 spectrometer at a frequency of 18.94 MHz at room temperature referenced to external dimethyl selenide.

2,2,5,5-Tetramethyltetrahydrofuran-3-one (1).

2,2,5,5-Tetramethyltetrahydrofuran-3-one (**1**) was synthesized from 2,5-dimethyl-3-hexyn-2,5-diol (Fluka AG, practical) according to Dupont [16-18]. The purity of the colourless liquid, bp 149° (lit bp 149° [16]), was checked by gc. The ¹H nmr (60 MHz carbon tetrachloride): δ 1.18 (s, 6H, CH₃), 1.32 (s, 6H, CH₃), 2.40 (s, 2H, CH₂); ir (carbon tetrachloride): 1750 cm⁻¹ (C=O); uv (absolute ethanol): λ max 298 nm (ϵ 30), 212 nm (ϵ 302); (cyclohexane): λ max 320 nm (ϵ 13), 310 nm (ϵ 25), 300 nm (ϵ 28), 213 nm (ϵ 317).

2,2,5,5-Tetramethyltetrahydro-3,4-furandione (2).

The dione **2** was formed by selenium dioxide oxidation of **1** in wet refluxing 1,4-dioxane (10 hours) [19] in yields of between 65 and 75%. The crude product was purified with several sublimations under reduced pressure (10 mm Hg) at ca. 50° to produce raspberry red crystals, mp 57° (closed capillary), (lit 54-56° [19]); ¹H nmr (60 MHz carbon tetrachloride): δ 1.32 (s, 12H); ¹H nmr (60 MHz deuterium oxide): δ 1.27 (s, 6H), 1.37 (s, 6H); ¹³C nmr (25.15 MHz deuteriochloroform): δ 25.5 (4 CH₃), 78.4 (2 quaternary C), 205.3 (2 C=O); ir (10% carbon tetrachloride): 1760, 1775 (sh) cm⁻¹ (C=O); uv (acetone): λ max 516 nm (CO-CO) (ϵ 46.5); (1,4-dioxane): λ max 516 nm (CO-CO) (ϵ 43.6); (absolute ethanol): λ max 322 nm (CO of the monohemiacetal form) (ϵ 48); (water): λ max 313 nm (CO of the dihydrate form) (ϵ 28.8); (aqueous sodium hydroxide solution): λ max 328 nm (CO of the hydrate anion) (ϵ 132).

2,2,5,5-Tetramethyltetrahydro-3,4-furandione Dihydrate (3).

When exposed to air the red dione **2** changed into a white dihydrate **3**, mp 75° (lit 74-75° [19]). The dihydrate consists of an hydrate and a gem-diol, and it can be dehydrated easily by melt-

ing and by sublimation; ¹H nmr (60 MHz deuterium oxide): δ 1.28 (s, 6H), 1.38 (s, 6H); ¹H nmr (60 MHz deuterioacetone): 1.22 (s, 6H), 1.28 (s, 6H), 2.88 (water of crystallization), 5.42 (2H, gem-diol), 1.38 (s, 12H, 4 methyl groups of the diketone form); ir (potassium bromide): 3520 and 3540 cm⁻¹ (OH), 1768 cm⁻¹ (C=O), 1640 cm⁻¹ (water molecule); uv (absolute ethanol): λ max 322 nm (ϵ 52.6); (water): λ max 313 nm (ϵ 30.6).

2',2',5,5,5',5',7,7-Octamethyl-2',5,5',7-tetrahydrofuro[3,4-d]-1-oxa-3,4-diselenin-2-spiro-3'-furan-4'(3'H)-one (4).

A mixture of **1** (5.0 g, 0.035 mole) and selenium dioxide (8.5 g, 0.077 mole, freshly sublimed) in ethanol (50 ml, 95%), was refluxed for 5 hours. The reduced selenium was removed at room temperature by a sintered glass funnel, washed with hot ethanol and dried to give 1.14 g of selenium. During the distillation of ethanol another batch of selenium (0.81 g) was removed (Se = 1.95 g). Ethanol was removed finally under reduced pressure until it began to sublime. The residue was heated to reflux with light petroleum (bp 40-65°), filtered and the crude product was obtained by room temperature evaporation of the solvent. Fractional sublimation *in vacuo* gave at 55-70°/10 mm Hg, 4.35 g (~80% of theory) of the dione **2**. The diselenide fraction was obtained at 75-105°/10 mm Hg as bright yellow crystals, 1.05 g (~14%), mp 97-104°. Repeated sublimations gave mp 104-106°. The sublimation residue (5.28 g) was selenium dioxide according to mass spectrometric analysis; ¹H nmr (200 MHz deuteriochloroform): δ 1.32 (s, 3H, CH₃), 1.35 (s, 3H, CH₃), 1.39 (s, 6H, 2 CH₃), 1.43 (s, 3H, CH₃), 1.49 (s, 3H, CH₃), 1.57 (s, 6H, 2 CH₃); ¹³C nmr (50.29 MHz deuteriochloroform): δ 25.54 (1 CH₃), 26.23 (1 CH₃), 26.87 (1 CH₃), 27.70 (1 CH₃), 28.42 (1 CH₃), 29.57 (2 CH₃), 31.28 (1 CH₃), 78.88, 81.38, 84.75, 87.23, 87.32 (spiro-C), 98.74 (=C-Se), 153.23 (O-C=), 203.98 (C=O); ⁷⁷Se nmr (18.94 MHz deuteriochloroform): δ 212.53, 316.48; ir (10% deuteriochloroform): 3030 cm⁻¹ (=C-H), 1740 cm⁻¹ (C=O), 1650 cm⁻¹ (C=C); uv (absolute ethanol): λ max 326.5 nm (ϵ 1000), 254 nm (sh ϵ 1100), 220 nm (ϵ 5400); (cyclohexane): λ max 328 nm (ϵ 1430), 254 nm (sh ϵ 1570), 219 nm (ϵ 7414); ms: m/z 440 (M⁺ 15%), C₁₆H₂₄O₄Se₂ (Found: 440.0007. Calcd. 440.0005, diff. 0.2 mmu), 425(15), 360(7), 285(31), 220(8), 140(75).

Crystal Structure Analysis of 4.

A bright yellow crystal of dimensions 0.3 x 0.4 x 0.4 mm was sealed in a capillary tube and used for X-ray measurements. Crystal data were measured at room temperature (18°) on a Nicolet P3F single-crystal diffractometer using graphite monochromatized MoK radiation (0.71069 Å). The cell parameters were determined by least-square treatment of the adjusted angular settings of 25 reflections (9 < 2 < 24.5). The structure was solved by direct methods. Successive Fourier calculations and full-matrix least-squares refinement with non-hydrogen atoms anisotropic and hydrogen atoms fixed isotropic, led to final R = 0.048 and RW = 0.053. The main crystallographic data and the structure determination conditions have been summarized in Table V.

Acknowledgments.

Financial support from the Emil Aaltonen Foundation and the Finnish Academy is gratefully acknowledged.

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